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Engle, Raymond Keeler; Pederson, Duane H.

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DESIGN AND CONSTRUCTION OF INSTRUMENTATION FOR INVESTIGATING THE OPTICAL DISPERSION OF AMMONIA GAS

Raymond Keeler Engle

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DESIGN AND CONSTRUCTION OF INSTRUMENTATION FOR INVESTIGATING THE OPTICAL DISPERSION OF AMMONIA GAS

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Raymond K. Engle

and

Duane M. Pederson

DESIGN AND CONSTRUCTION OF INSTRUMENTATION FOR INVESTIGATING THE OPTICAL DISPERSION OF AMMONIA GAS

by

Raymond Keeler Engle
Lieutenant Commander, United States Navy

and

Duane Melvin Pederson
Captain, United States Army

Submitted in partial fulfillment
of the requirements
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IN

PHYSICS

from the

United States Naval Postgraduate School

PREFACE

This investigation was conducted at the U.S. Naval Postgraduate School, Monterey, California, during the period from 2 January 1956 to 20 April 1956.

The purpose of the investigation was to design and construct a gas refractometer for the determination of the index of refraction of gases in the visible region of the electromagnetic spectrum, and to determine the refractive index of ammonia gas within the range of the system.

Our apparatus consisted of a gas admission system, a double chamber refraction tube, an evacuation system, and a double slit interferometric optical system, using a mercury arc and a white light source, coupled with a monochromator, as an electromagnetic source.

Two sets of data for the index of refraction of ammonia gas are published in the International Critical Tables. (2) It was our intention to provide new data to resolve the difference.

The writers wish to express their appreciation to Professor Sydney H. Kalmbach for the suggestion of this topic and for his guidance and encouragement; to Mr. M.K. Andrews and Chief Opticalman R.C. Moeller, U.S. Navy, for their skillful and highly cooperative assistance in our construction phase; and to Mr. H.S. Perry for machining done on the refractometer tube.

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LIST OF ILLUSTRATIONS

TABLE OF SYMBOLS

E	Electric field
F	Force
K	Dielectric constant
L	Length of refraction tube
N	Number of atoms per unit volume
P	Dipole moment per unit volume
$\triangle P$	Pressure differential produced between chambers of the refraction tube
S_n	Slit
T	Temperature, telescope
c	Velocity of light in free space
e	Charge on one electron
g	Quasi-frictional constant
m	Mass of one electron
m_d	Dipole moment per atom
$\triangle m$	Fringe count
n	Index of refraction
q	Charge
v	Velocity
α	Absorption coefficient
β	Imaginary part of propagation constant
γ	Propagation constant
ϵ	Permittivity
ϵ_o	Permittivity in free space
λ	Wavelength

ν	Frequency of electromagnetic radiation
ν_o	Frequency of electron vibration
χ	Susceptibility
ω	Angular velocity

CHAPTER I

INTRODUCTION

1. Summary

The object of our investigation was twofold. Our primary interest was to design and construct a simple type of interference refractometer for measurement of the refractive index of gases in the visible region of the electromagnetic spectrum. Our second problem was to determine the index of refraction of a gas within the range of the apparatus.

The design and construction phase of this work was directed toward building a gas refractometer containing the minimum components necessary to achieve the optimum accuracy and ease of operation commensurate with the time available for the investigation. Detailed descriptions of the components used are contained in Chapter II of this report.

In the second phase, ammonia gas was selected for the refractive index determination. In the investigation it was found necessary to devise suitable laboratory techniques for use with our apparatus, which are discussed in Chapter III. A complete evaluation of the results is contained in Chapter IV.

2. Theory of Dispersion in Gases

The existance of an index of refraction in gases is caused by its electric dipole moment per unit volume. In the case of polar gases, such as ammonia, the total dipole moment is the sum of the induced and permanent dipole moments.

The induced dipole moment per electron is proportional to the applied electric field, or $m_d = E$, where the constant of proportionality is called polarizability. The contribution to the index of refraction of a gas by this effect is derived in the Appendix.

Permanent dipole moments are exhibited in materials containing polar molecules. Ammonia contains three hydrogen atoms with a center of positive charge, which is balanced by the negative charge of the nitrogen atom, as shown in Figure 1. The hydrogen end of the molecule is positively charged and the

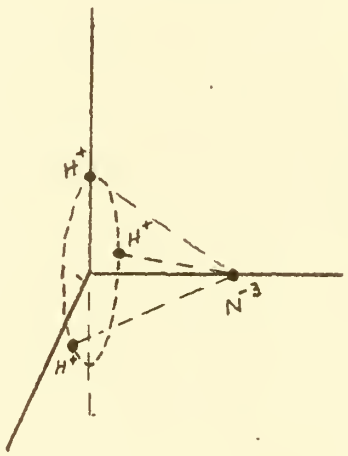

Figure 1. Structure of Ammonia Molecule

nitrogen end is negatively charged. In the absence of an external field, the molecules in the gas will be oriented in random directions, so that even though each molecule has a moment, the average moment per unit volume will be zero. An impressed field, however, tends to orient the molecules, and there is a net dipole moment resulting which is proportional to

the impressed field. In our case the impressed field is the E vector of the electromagnetic wave from the light source. The orientation of these permanent dipoles is opposed by temperature agitation, and by kinetic theory it can be shown that the resulting polarization in a given external field is inversely proportional to the absolute temperature(3).

3. Determination of Refractive Index by Interference Measurements.

The refractive indices of a gas are the ratios of the wave velocity of light in vacuum to the velocities in the gas. The actual measurement of these indices can be done in several ways, but some type of interference refractometer is the most accurate and the most convenient instrument for measurements on gases which have small indices(1). The basic requirement is that the arrangement provide a means for dividing a beam of light into two or more parts which travel different optical paths and then recombine to form interference fringes. The optical arrangement to be used here in the discussion of the refractive index determination is a form of Rayleigh's refractometer and is illustrated in figure 2.

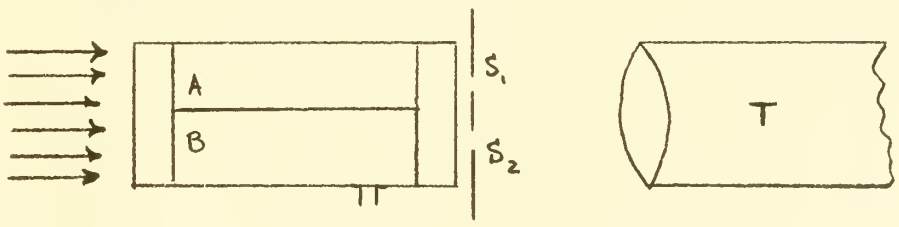

Figure 2. Light Paths in a Rayleigh Type Gas Interferometer.

The refraction tube consisting of two tubes, A and B, is closed on each end with a good glass window and placed in the path of a monochromatic parallel beam so that light incident on slit S_1 traverses tube A and that on slit S_2, tube B. The two beams are then brought together again in the focal plane of the telescope T, to interfere and produce measurably observable interference fringes.

In order to measure the index directly with respect to a vacuum, rather than to compare with a secondary standard, both tubes are initially evacuated. Tube A remains evacuated while the gas being investigated is introduced gradually into the path of the light traversing tube B. By admitting gas to tube B, the density is changed and therefore the optical path of one of the interfering beams is altered, causing a change in position of the fringes. Technically it is said that there is a shift of fringes because on viewing them, the central fringe displaces toward the side of the higher-index medium. By observing the number of fringes which pass the station previously occupied by the central fringe, the computation of the refractive index for the gas being investigated is made possible by using the following equation[5]:

$$n-1 = \Delta m \cdot \lambda \cdot \frac{T}{273} \cdot \frac{760}{\Delta P} \cdot \frac{1}{L}$$

This equation is based on the difference in optical path for the parallel beam of light and includes the correction factors necessary to convert the determination to standard

temperature and pressure. The change in density necessary for the computation is obtained by observing the change in pressure in tube B during the fringe shift. It is worthy of note that temperature is obviously an important factor because heat changes the density of the gas and consequently its refractive index.

CHAPTER II

DESIGN AND CONSTRUCTION OF APPARATUS

1. General Description of Instrumentation.

As shown in figures 3 and 5, our refractometer consisted of the following components:

a. A double gas admission system with liquid nitrogen traps for gas purification and collection.

b. A two chambered refraction tube to contain the standard and the gas under investigation.

c. A temperature measuring device to measure the temperature of the gas in the refraction tube. This consisted of a copper-constantan thermocouple connected to a sensitive Rubicon bridge assembly, providing accuracy to one-tenth of a degree Centigrade.

d. A mercury manometer to measure the pressure differential between the two chambers of the refraction tube. A cathetometer was used to read the manometer level, which provided accuracy to one-tenth of a millimeter.

e. An evacuation system containing a vacuum pump, capable of pumping a vacuum of one-thousandth of a millimeter of mercury, to create a vacuum, and a Pirani gauge to measure the vacuum.

f. An optical system consisting of a mercury arc, a white light source, a monochromator coupled with a collimator, a double slit, and two seven power telescopes in tandem for magnification of the double slit interference pattern.

g. A Cyclotron counter, wired to a telegrapher's key and a 45 volt battery, was used as a recording device in fringe counting.

h. A steel frame optical bench, on which the refraction tube and the optical system were mounted.

2. Gas Admission and Evacuation System

The first requirement of this system was to obtain a vacuum of the order of one-thousandth millimeter of mercury. Also, it was necessary to hold this vacuum to a rise of less than one-tenth millimeter of mercury for the duration of a determination, with the vacuum pump closed off. One-tenth millimeter was the limit of accuracy of our manometer measurements. All stationary components of this system were made of pyrex glass. This made it possible to effectively locate all leaks by passing an ungrounded high potential electrode of a spark coil over the surface of the glass[7].

The problem of gas disposal was resolved by installing a liquid nitrogen trap with a detachable outer cylinder ahead of the vacuum pump. This froze the gas which was drawn off the system into the outer cylinder of the trap, where it was readily disposable without contaminating the air in the laboratory.

Liquid nitrogen traps were installed in the system to provide a means of purifying the gas and a means of admitting the gas slowly enough to make accurate fringe counting

possible. The method used is discussed in detail in Chapter III.

It was necessary to devise a means of evacuating both sides of the system simultaneously or individually. This was required in order to get the best vacuum possible in both sides of the system before starting a series of runs, and also to insure that the vacuum side of the system was not contaminated by gas between individual runs.

The mercury manometer, the copper-constantan thermocouple, and the Pirani gauge, previously mentioned, were installed as shown in Figure 3.

3. Refraction Tube

The experimental refraction tube used to contain the gas under investigation was constructed from a piece of a 5/8 x 1 inch rectangular brass wave guide. Two sections of approximately 75 centimeters in length were soldered together to form a two chambered tube and then fitted at each end with a collar arrangement. The collars, figure 4, were machined from brass stock and tinned to the tube. The end faces of the collars were made absolutely parallel to each other by means of a lathe so that distortion of optical path would be kept to a minimum. A channel was cut on each face to facilitate the use of a neoprene rubber gasket in helping to seal the ends and also in helping to keep the two chambers separated from each other with respect to the evacuation and gas admission system. To seal the ends of the refraction

tube, two three-inch diameter, one-half inch thick, high quality homogeneous optical disks with parallel plane surfaces were used. Each glass disk was held in position by a cover plate bolted to the collar and was pulled vacuum-tight against the rubber gasket by the vacuum created in the assembled apparatus.

The refraction tube was connected to the evacuation and gas admission system by means of two brass fittings, one for each chamber, into which could be inserted the glass tubing. To insure a vacuum-tight seal, these fittings were tinned to the refraction tube and the glass sealed in them with Apiezon "W" wax. Finally, to help us achieve and maintain a good fore-pump vacuum, the refraction tube was dipped in glyptal before final assembly, dried and then baked until the coating was hard. It was then mounted by means of brackets to a steel frame optical bench as shown in Figure 5.

4. Optical System

Our optical system was designed to give us a measurably observable interference pattern. The components used to achieve this were a monochromator, a collimator, a double slit and two telescopes. Their arrangement is as shown in Figure 5.

The Gaertner glass monochromator used was calibrated, prior to use, against the emission line spectra produced by helium and hydrogen Geissler tubes and a mercury arc. The

exit slit of the monochromator was then adjusted to allow only a narrow band of the wave length corresponding to the drum setting to pass through it. Next, careful alignment of the monochromator on the optical bench positioned the exit slit in the focal plane of the collimator. The collimator in turn was adjusted so that the emerging monochromatic parallel beam of light would be split by the refraction tube into two beams of approximately the same intensity. The double slit, cut from a manila folder, was then carefully positioned on the end window of the refraction tube nearest the telescope so that the light traversing each chamber passed through a different slit. The width of the wall between the two chambers determined the width of the slits and the distance between them necessary for obtaining an optimum interference pattern. The telescopes used were one from a Gaertner spectrometer and one from a pair of 7x50 binoculars. The binocular telescope was added in tandem primarily for its magnification, but also for its field of view and light gathering power. To further aid in making an accurate fringe count, a slit was fashioned from a razor blade and mounted on the inside of the ocular of the binocular. The width of this slit was so adjusted that it included only one bright fringe when observing the interference pattern. This in effect isolated one fringe from the interference pattern and made accurate visual counting possible.

CHAPTER III

EXPERIMENTAL PROCEDURE AND RESULTS

1. Operating Procedure

During the investigation of ammonia, it was found necessary to devise suitable laboratory techniques for use with our apparatus. In our preliminary work, a number of runs were made on nitrogen gas to test the apparatus. From these runs, the following operating procedure and laboratory techniques were devised and were used for each experimental run, in the order listed:

a. Both sides of the refraction tube and the evacuation and gas admission systems were evacuated and checked for leaks by a rate-of-rise measurement made with the Pirani gauge. If the vacuum held, a dewar flask of liquid nitrogen was placed on the trap preceding the vacuum pump and the system was again pumped down to the best vacuum.

b. With the vacuum side of the system closed off, the gas side of the system was filled and evacuated several times with the gas being investigated. This helped to reduce the probability of impurities being in the gas side to a minimum.

c. Immediately after the last flushing sequence, a dewar flask of liquid nitrogen was placed on the gas admission trap. As the gas was admitted, it was frozen in the bottom of the trap until there was enough for several runs.

d. With the desired light source illuminating the entrance slit of the monochromator, a wave length was selected and the proper setting made on the monochromator drum. A check of the interference pattern was then made for positioning the binocular telescope so that a dark fringe was centered in the field of view.

e. The manometer level was then measured on the vacuum side by means of the cathetometer. This side was chosen because the meniscus always remained in the same shape, whereas the meniscus on the gas side was observed to flatten when the gas was admitted.

f. With the pump closed off, the nitrogen flask was then removed from the gas admission trap and the frozen ammonia was allowed to melt and evaporate. Because of the temperature differences involved, this process proceeded slowly and could be controlled.

g. The temperature of the gas was measured at the beginning and end of each run by means of the thermocouple; the room temperature was also recorded.

h. The movement of fringes toward the gas side of the system was counted by means of the cyclotron counter and the number recorded.

i. After a sufficiently long count, 110 to 160, the gas admission was stopped by closing off the trap containing the melting ammonia from the system. The manometer level was then read again on the vacuum side of the system and the

result recorded. The pressure differential between the vacuum and gas sides of the system is equal to twice the difference of the observed pressure readings.

j. The gas was then collected in the gas admission trap again by placing the liquid nitrogen flask back on the trap. When most of the gas was collected the gas side was again opened to the vacuum pump. This was done to make certain that the mercury in both tubes of the manometer were at the same level. During the entire run, the vacuum side was closed off from the pump and the gas side. When needed, as shown by a plot of rate-of-rise measurement versus time, the vacuum side was evacuated.

k. It is obvious that two people are required for this procedure: one to count the fringes, the other to make the temperature and pressure measurements and observe the gas admission. The pressure could be read precisely each time by reflecting the beam of a flashlight from the manometer tube. This gave more consistent pressure measurements because the top of the meniscus was clearly indicated.

2. Results

The apparatus as designed and assembled is capable of measuring the refractive index of a gas to a precision of less than one percent for any given wave length. This is easily verified by considering the effect of an error in measurement of the quantities used in the refractive index determination.

All computations were made using the formula

$$n-1 = \Delta m \cdot \lambda \cdot \frac{T}{273} \cdot \frac{760}{\Delta P} \cdot \frac{1}{L}$$

but by rearranging the terms and differentiating the function, the following expression represents the precision capabilities of the apparatus:

$$dF = \frac{d(\Delta m)}{\Delta m} + \frac{dT}{T} - \frac{d(\Delta P)}{\Delta P} - \frac{dL}{L} \quad \text{where } F = \frac{\Delta m T}{\Delta P L}$$

and $\frac{760\lambda}{273}$ is a constant for any given wave length.

Based on the characteristics of the measuring devices already mentioned elsewhere in this report, evaluation of the effects of an error in measurement are then as follows:

$$\frac{d(\Delta P)}{\Delta P} = \frac{.4}{200} \text{ (maximum) } = 0.2\% \text{ (average)}$$

$$\frac{dT}{T} = \frac{.1}{295} = 0.034\%$$

$$\frac{dL}{L} = \frac{.1}{74.6} = 0.134\%$$

$$\frac{d(\Delta m)}{\Delta m} = \frac{1}{125} = 0.8\%$$

Therefore, $dF = 0.8 + 0.034 - 0.2 - 0.134 = 0.5\%$

The dispersion curve for ammonia gas in the visible region of the electromagnetic spectrum, as determined by this investigation, is shown in Figure 6. The values used for plotting this curve are found in Table 1. In determining the tabulated values for n - 1, several runs were made for each wave length and a statistical mean and standard deviation

evaluated for each point. The runs used for each wave length include runs from different gas samples from the gas bottle and measurements made by both authors. It should also be mentioned at this time, that the ammonia gas used was anhydrous ammonia bottled by the Matheson Co., Inc., Joliet, Illinois. The minimum guaranteed purity was published to be 99.5%. It is conceivable that the refractive indicies attributed to any of the possible one-half percent impurities are of the same order of magnitude as that of the ammonia; therefore, it is felt that any variation in our final results by impurities from the bottled gas is negligible.

Table 1. Calculated Data for the Refractive Indicies of Ammonia at 0°C, 760 mm Hg

λ (Å)	$(n-1) \times 10^{-7}$
4861.33	3849
5209.1	3819
5460.74	3796, 3797*
5650	3792
5790.66	3786, 3787*
5875.62	3785
6110	3780
6438.47	3768
6562.79	3766

*For Mercury Emission Line Spectra

CHAPTER IV

EVALUATION OF DATA AND CONCLUSION

1. Comparison with Existing Data

For comparison, the values of the indicies of refraction in the visible spectrum were taken from the "International Critical Tables" [2] and are presented in Table 2 together with those determined by this investigation.

Table 2. Refractive Indices of Ammonia at 0°C., 760 mm. Hg.

	$(n-1) \times 10^{-7}$		
λ (Å)	Fricke	Engle-Pederson	Richards
4861.3	--	3849	3918
5209.1	3800	3816	--
5460.7	3786	3795	3870
5790.7	3770	3786	--
5875.6	--	3783	3848
6438.5	3746	3768	--
6562.8	--	3766	3826

Since our data falls between that which is published, confirmation as to the validity of our data is indicated because there are no true values.

2. Correlation of Results with Dispersion Theory

As a check on the dispersion curve obtained from this investigation, the index of refraction of ammonia was computed for various wavelengths, using the formula derived in

Appendix I. These computed values were compared with our experimental data, as shown in Table 3. Although A and ν_0 were determined empirically from experimental data, the fact that good agreement exists all along the curve is an indication of accuracy in a relative sense.

Table 3. Comparison of Observed and Computed Indices of Refraction for Various Wavelengths.

λ (cm)	Observed n	Computed n
4.8×10^{-5}	1.0003856	1.0003850
5.2×10^{-5}	1.0003817	1.0003817
5.6×10^{-5}	1.0003793	1.0003796
6.0×10^{-5}	1.0003779	1.0003779
6.4×10^{-5}	1.0003769	1.0003766
6.8×10^{-5}	1.0003762	1.0003759

3. Suggestions for Improvement and Further Utilization of the Apparatus.

The degree of precision of our apparatus could be improved somewhat by using a photoelectric cell and an electronic counter to count the fringes. This would result in greater precision by eliminating human error and by making it possible to take longer counts.

Two suggestions for further utilization of the apparatus: First, it would be possible to extend the range into the infrared region of the electromagnetic spectrum by replacing the glass windows of the refraction tube with suitable window material for the infrared region, such as potassium bromide.

Second, the dispersion curve of hydrogen chloride gas would provide an opportunity to accurately locate an absorption band which exists within the very near infrared region of the spectrum. This could be accomplished using the glass windows which are presently installed.

APPENDIX

DERIVATION OF REFRACTIVE INDEX FORMULA

The equation of motion for an electron under the force of the electric field due to electromagnetic radiation is

$$m(d^2x/dt^2) + mg(dx/dt) + m\omega_o^2 x = eE\, e^{j\omega t} ,$$

where the first, second, and third terms on the left are inertia, friction, and restoring force, respectively, and the term on the right is the force of the electric field.

The force due to the magnetic field in the wave is negligible due to the factor v/c in the basic force equation $F = q(E+\vec{v}/c \times B)$.

Solution of the equation of motion by letting $x = x\, e^{j\omega t}$ is

$$x = \frac{(e/m)E}{(\omega_o^2 - \omega^2 + j\omega g)}$$

$$m_d = ex$$

$$P = Nex \quad \frac{N(e^2/m)E}{(\omega_o^2 - \omega^2 + j\omega g)}$$

$$K = 1 + \chi_e = 1 + P/E\epsilon_o = 1 + \frac{N(e^2/m\epsilon_o)}{(\omega_o^2 - \omega^2 + j\omega g)}$$

Considering the z direction, the direction of propagation, the electromagnetic wave equation with respect to the field E, considering σ equal to zero and μ equal to one, is

$$\partial^2 E/\partial z^2 - \epsilon\, \partial^2 E/\partial t^2 = 0$$

Let $E = E\, e^{j\omega t - \gamma z}$

The solution to the electromagnetic wave equation is then

$$\gamma = j\omega\sqrt{\epsilon_o}$$

If we put γ in complex notation

$$\gamma = \alpha + j\beta$$

Then $E = E\, e^{-\alpha z}\, e^{-j(\omega t - \beta z)}$

Here the $e^{-\alpha z}$ term becomes one, because α, the abosorption coefficient, is considered to be zero.

Since $e^{-j(\omega t - \beta z)} = \cos(\omega t - \beta z)$ + an imaginary term.

Therefore $v = \omega/\beta = \omega/\omega\sqrt{\epsilon} = 1/\sqrt{\epsilon}$

also $c = 1/\sqrt{\epsilon_o}$

Since $n = c/v$

$$n = \sqrt{\epsilon}/\sqrt{\epsilon_o} = \sqrt{K}$$

It then follows that

$$n = 1 + \frac{N(e^2/m\epsilon_o)}{(\omega_o^2 - \omega^2 + j\omega g)}$$

$$= 1 + \frac{N(e^2/m\epsilon_o)}{2(\omega_o^2 - \omega^2 + j\omega g)} + \text{negligible higher order terms}$$

$$n - 1 = \frac{N(e^2/m\epsilon_o)}{2(\omega_o^2 - \omega^2 + j\omega g)}$$

If we let $A = \frac{N(e^2/m\epsilon_o)}{8\pi}$ and consider $(\omega_o^2 - \omega^2) >> j\omega g$

Then $n - 1 = \frac{A}{(\nu_o^2 - \nu^2)}$

The constants A and ν_o were evaluated from the experimentally

determined dispersion curve of the gas under consideration. Knowing these values, the formula was then used to compute a dispersion curve, which when plotted checked the shape of the experimentally determined dispersion curve.

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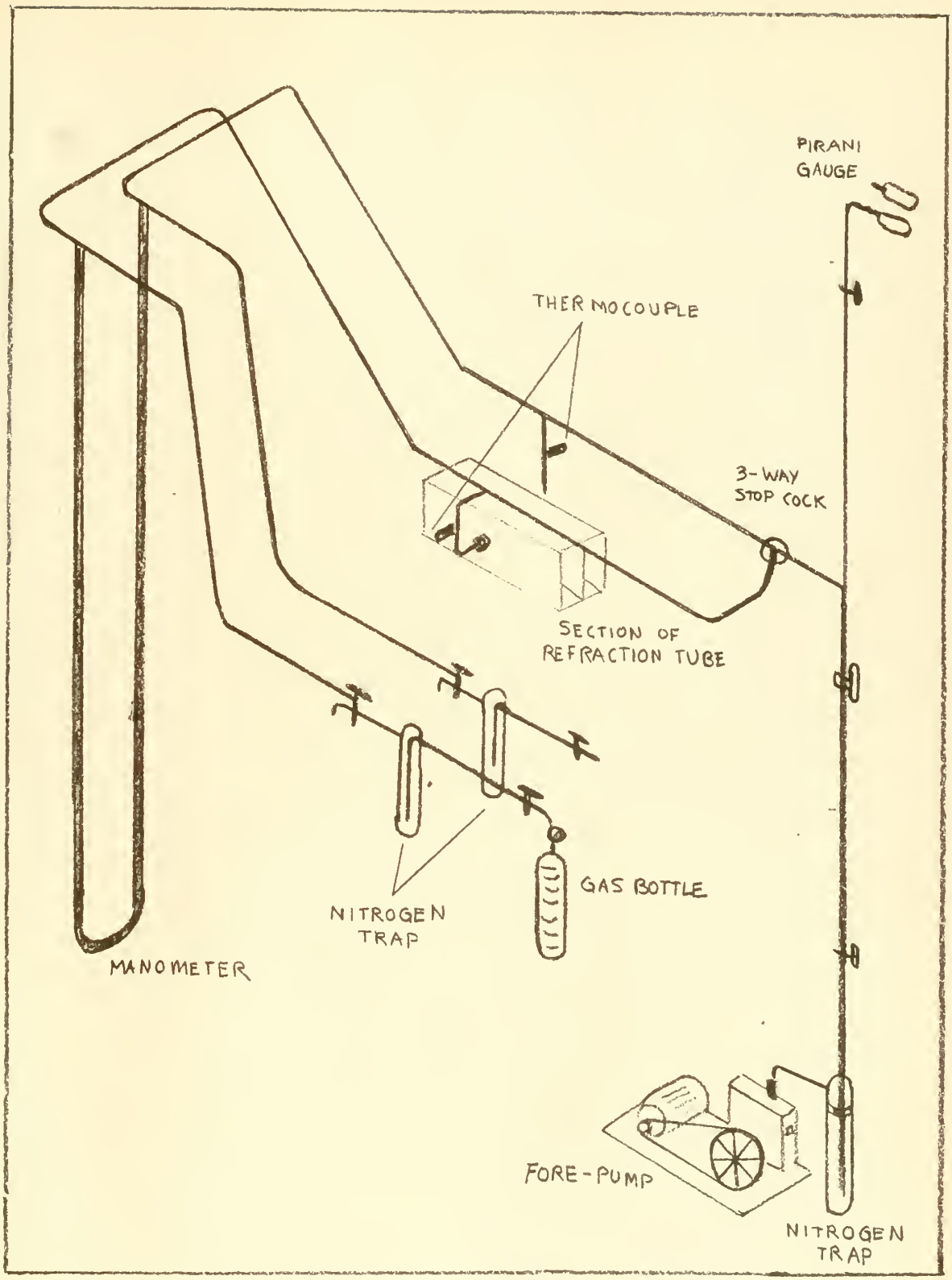
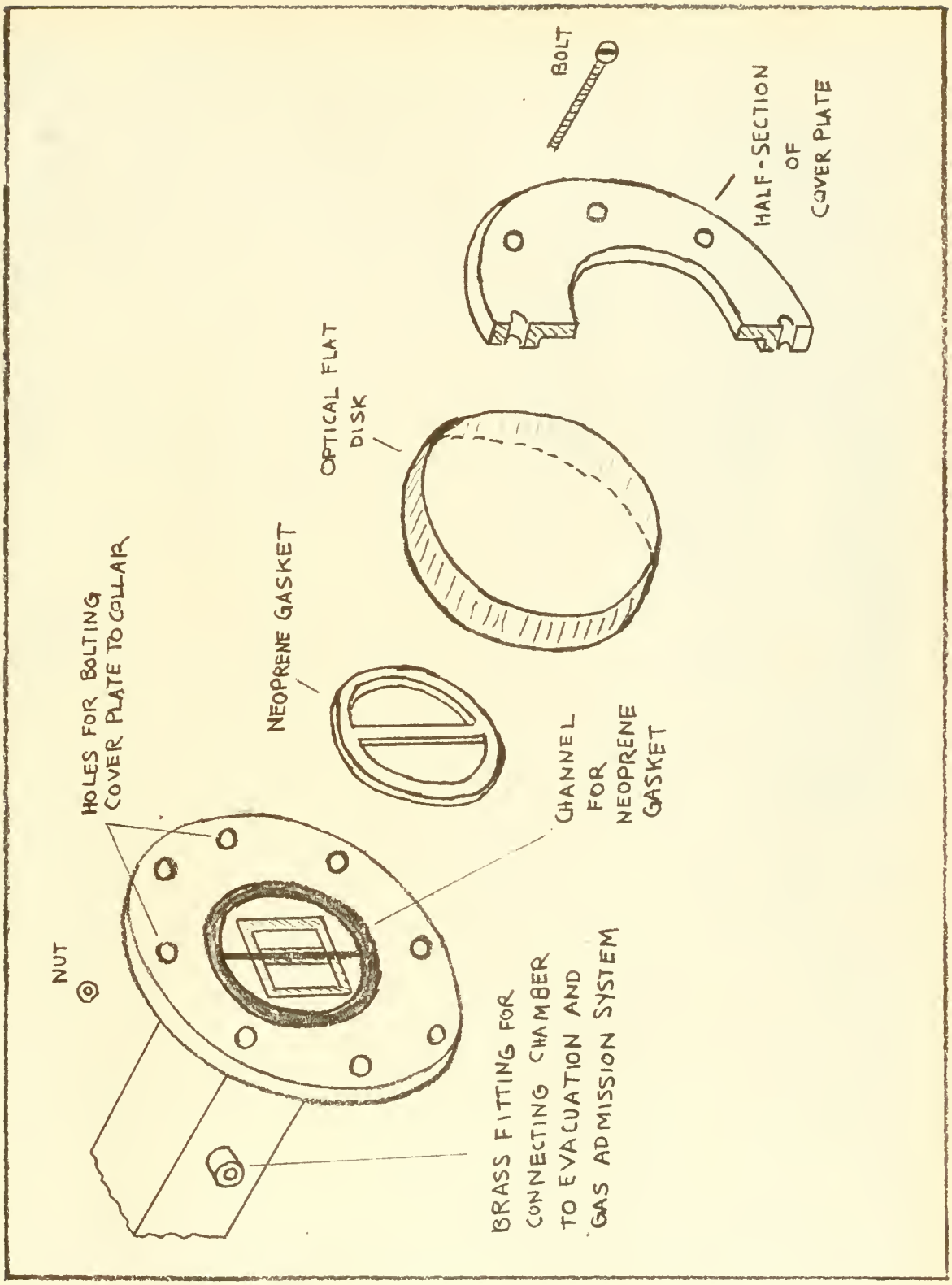
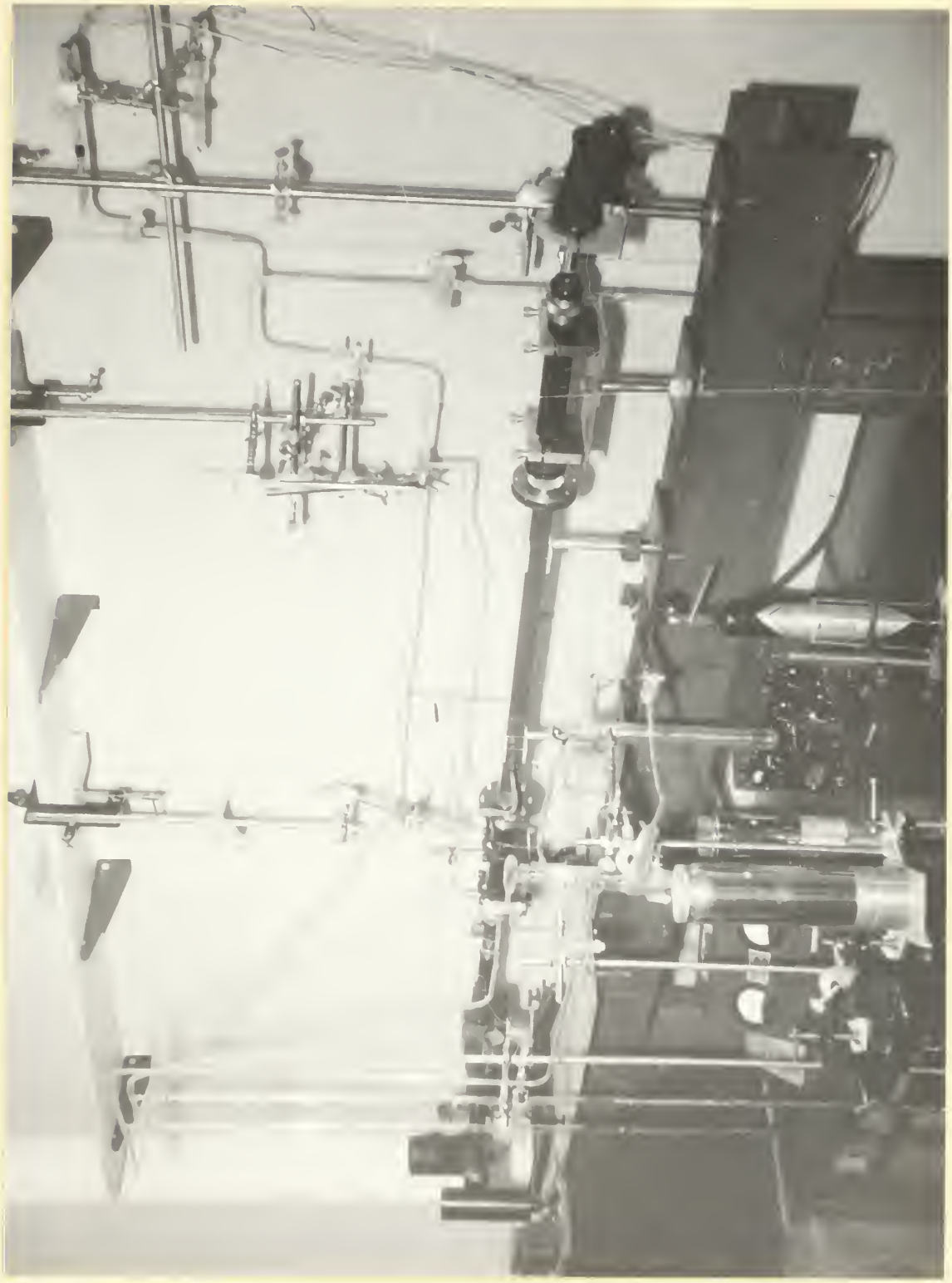


Figure 3. Gas Admission System



Refraction Tube Collar Assembly

Figure 4.



Photograph of Apparatus

Figure 5.

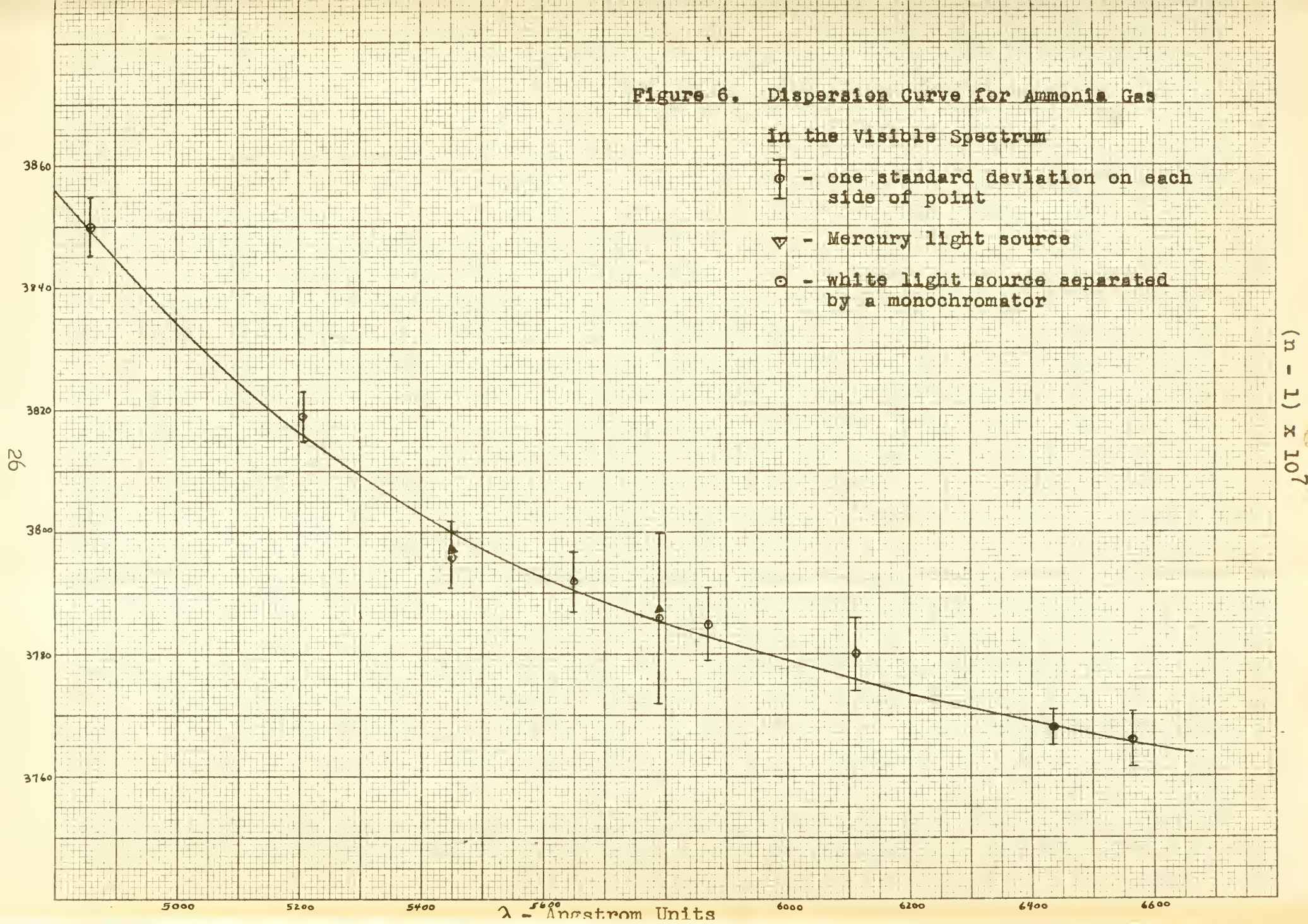


Figure 6. Dispersion Curve for Ammonia Gas
in the Visible Spectrum
- one standard deviation on each side of point
- Mercury light source
- white light source separated by a monochromator
3860
3840
3820
3800
3780
3760
(n - 1) x 10^7
5000
5200
5400
5600
6000
6200
6400
6600
λ - Angstrom Units

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